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EXTRACTION AND USES OF BERYLLIUM IN GERMANY

BY

GEORGE T. MOTOCK

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By George T. Motock[2]

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FOREWORD

The author prepared the following report as the result of a trip to Germany in the summer of 1945 for the Technical Industrial Intelligence Commitee Subcommittee on Metals and Minerals. The purpose of this committee, which functions under the Joint Intelligence Committee of the Joint Chiefs of Staff of the United States Armed Forces and of which the Bureau of Mines

1/ The Bureau of Mines will welcome reprinting of this paper provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7357."

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is a member, was to obtain information of interest to American Industry and of value to the United States Joint Control Council for Germany. This paper was submitted through the Joint Intelligence Objectives Agency and was released for publication through the Bureau of Mines in order to reach the audience that can make the best use of it. In all cases the individuals or teams sent on the investigating missions were chosen from the best-qualified men available in the industry and in the various Government agencies. The material herein was prepared entirely by the author, who is not connected with the Bureau of Mines.

SUMMARY

The extraction of beryllium and its salts in Germany has been handled by the units of Degussa (Deutsche Gold und Silber Scheide Anstalt) in Frankfurt am Main and at Rheinfelden am Rhein near Basel.

Beryl ($3BeO.Al_2O_3.6SiO_2$) was the chief beryllium ore used in the process. This ore was mainly imported from South America through Portugal, France, and Italy. No important beryllium-ore deposits are found in Germany, but they do occur in Rumania, where they are considered to be easily mined.

Degussa has many units (all scattered in the plant at 215 Guteleutestrasse, Frankfurt) for making different beryllium salts or intermediate products before electrolysis of the metal in five double-unit cells. In Rheinfelden, Degussa has a new plant - 90 percent completed - for making beryllium salts and beryllium metal (one double-unit cell).

Degussa sold most of its beryllium oxide and beryllium metal to Heraeus Vacuumschmelze at Hanau near Frankfurt, where beryllium alloys for watch and instrument springs were made and shipped to Switzerland for sale to the United States watch industry.

Heraeus Vacuumschmelze also made copper-beryllium alloys by the reduction, in an arc electric furnace, of beryllium oxide with carbon in the presence of powdered copper or copper oxide. Heraeus Vacuumschmelze is a unit of the large Siemens-Halske concern in Berlin.

Many firms in Germany were interested in beryllium, as indicated by the patents obtained by I. G. Farbenindustrie, Siemens-Halske, Heraeus Vacuumschmelze, and Deutsche Gold und Silber Scheide Anstalt; later, even Metallgesellschaft was interested in beryllium.

INTRODUCTION

The information in this report has been obtained by personal visits to the offices and plants of companies dealing with the extraction and uses of beryllium.

Deutsche Gold und Silber Scheide Anstalt (Degussa)

At present, the main office is at 31 Guteleutestrasse, and the two plants are at 7 Weissfrauenstrasse and 215 Guteleutestrasse, all in Frankfurt am Main.

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Beryllium metal was produced at the old plant (215 Guteleutestrasse) in Frankfurt; however, a new plant for producing beryllium metal has been built (although only 90 percent completed) at Rheinfelden.

This establishment was investigated several times between July 27 and September 18, 1945, and the following members of the personnel have been interviewed:

Dr. Walter Völkel, expert on beryllium, 215 Guteleutestrasse, Frankfurt.
Dr. Gustav Jaeger, in charge of ceramics department, Stierstadt, near Frankfurt. He has several German patents on beryllium. He lives at 25 Taunusstrasse, Neu Isenburg, near Frankfurt.
Dr. Hans Medweth, in charge of the plant at Rheinfelden, where the new beryllium plant was being built.
Ernst Brach, foreman of beryllium plant, Rheinfelden.
Dr. Leopold Harant, chief chemist, Stierstadt, Frankfurt.
Dr. Martin, 31 Guteleutestrasse, Frankfurt.

Details of the organization, plants, products, etc., of Degussa are given in a report by the author, Lithium Extraction and Uses, prepared for the T. I. I. C. Metals and Minerals Subcommittee, to be issued by the Bureau of Mines as an information circular.

Heraeus Vacuumschmelze A. G.

This concern, which since 1936 has been part of Siemens-Halske A. G., Berlin, produces beryllium alloys in electric arc furnaces and vacuum induction furnaces. It was investigated several times from July 25 to September 10, 1945; the following members of the staff were interviewed:

Dr. Walter Deisinger, in charge of both plants at Hanau and at Sterbfritz near Frankfurt. He came to Hanau in February 1945 from Siemens-Halske, Berlin.
Dr. Hans Hiemens, chief engineer, who has been with Heraeus Vacuumschmelze for 20 years.
Dr. Herbert Gruber, chief metallurgist, was 24 years with the company and worked very closely with W. Rohn, inventor of vacuum melting.
Dr. Karl Schichtel, melting-shop superintendent.
Hans van der Au, works engineer, 25 years with Heraeus Vacuumschmelze.

EXTRACTION OF BERYLLIUM SALTS AND BERYLLIUM METAL

The chief beryllium ore is beryl ($3BeO.Al_2O_3.6SiO_2$), which contains about 12 percent BeO, 22 percent Al_2O_3, and 62 percent SiO_2. Although other beryllium ores, such as chrysoberyl ($BeO.Al_2O_3$) and phenakite ($2BeO.SiO_2$), were considered as sources of beryllium, these two ores are not as available as beryl. In southern Germany there are some insignificant and scattered beryl deposits, which do not represent an important source.

Most of the beryl used in the production of beryllium has been imported from South America (Argentina, Brazil) and from India through Portugas, France,

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and Italy; some comes from Norway and Finland and from France (Limoges Savoie) and Madagascar. Degussa was also considering importing some beryllium ore from Rumania, where, it is claimed, it can be easily mined.

Dr. Völkel reported that during 1940-43 Degussa imported:

Year	Amount and source of beryllium ore
1940	10 tons through Le Havre, France
1941	200 tons from South America on Italian ship to France
1942	60 tons from Alais Forges, Savoie-France
1943	500 tons from Chivasso, Italy

Beryllium Salts

At the Degussa plant in Frankfurt and at the new beryllium plant at Rheinfelden, the beryllium salts necessary to produce beryllium metal are processed as shown in the outline below (prepared by Dr. Völkel of Degussa) - see also the accompanying flow sheet:

Beryl is first ground in a ball mill to 800-mesh per square centimeter.

About 200 kg. (440 lb.) of finely ground beryl is mixed for one-half hour with 100 kg. (220 lb.) of burnt lime (CaO) in a drum.

This mixture of beryl and burnt lime, in 60- to 70-kg. (130- to 150-lb.) lots, is dropped in a rotary furnace, where it is sintered (or melted) at 1,400° to 1,500° C. (2,550° to 2,750° F.) during a 1½- to 2-hour melting period. This rotary furnace is gas-fired at the rate of 115 cubic meters (4,067 cubic feet) of gas per hour. For one melting period about 180 to 240 kg. (500 lb.) of sintered (melted) mixture is produced.

The furnace is lined with fire clay. Ten to 100 tons of mixture is melted (sintered) on one lining. In 1940 one lining lasted for 10 tons of mixture but later, by regulating the flame (making it larger and nonturbulent), 100 tons of mixture was sintered (melted) on one lining.

By this sintering process the lime ties up the silica from beryl, forming calcium silicates and beryllium compounds, which react with sulfuric acid more readily than raw beryl.

The molten (sintered) mixture is then dropped in a pan containing water, where the mixture breaks into small particles.

The wet sinter (melt) mixture is next dried in a rotary kiln, and then it is ground in a ball mill to a fineness of 800-mesh per square centimeter (about 75 meshes to the linear inch).

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Beryllium Sulfate

The finely ground melt (sinter) mixture is next sulfatized. Sulfatizing is done by taking 300 kg. (650 lb.) of melt mixture and mixing it in a sulfatizing pan with 480 kg. (1,000 lb.) of technical sulfuric acid and 40 liters of water in order to obtain 420 kg. (930 lb.) of sulfate mass. This sulfate mass consists of sulfates of beryllium, calcium, aluminum, and iron.

Silica Separation

Silica separation is obtained by taking 420 kg. (930 lb.) of sulfate mass and mixing it with 350 liters of wash water and 0.5 kg. (1 lb.) of glue and heating in a leaching kettle (with steam) to 105° C. (223° F.). The glue is added in order to flocculate the silica.

This mixture is then filtered through filter press No. 1 (nitrated cotton filter cloth) where silica (SiO_2) and calcium sulfate ($CaSO_4$) are retained.

The solution that passed through the filter press contains sulfates of beryllium, aluminum, and iron. The wash water is recirculated for further use.

Aluminum Separation

The solution, containing sulfates of beryllium, aluminum, and iron, is mixed with 75 kg. (160 lb.) of ammonium sulfate and crystallized on a pan at 100° C. (212° F.).

This is then cooled and centrifuged; the resulting solid product is ammonium-aluminum sulfate, thus eliminating aluminum.

Elimination of Iron

Iron is eliminated by taking 600 liters of solution containing sulfates of beryllium and iron and mixing with 1,200 liters of water and with 3 liters of hydrogen peroxide (H_2O_2) in order to oxidize the iron in an iron-removal kettle. To this is added 75 kg. (150 lb.) of calcium carbonate, which was previously mixed with water to a pH of 3.8 to 4.2.

The resulting mixture is filtered in filter press No. 2. In the filter press a residue is retained consisting of iron hydroxide and calcium sulfate; thus the iron is eliminated. The solution contains only beryllium sulfate.

Beryllium Hydroxide

The beryllium sulfate solution is precipitated in a precipitation kettle by adding ammonia gas (NH_3) to precipitate beryllium hydroxide. This is filtered through filter press No. 3 and washed. This beryllium hydroxide contains 6 to 7 percent of beryllium oxide.

The hydroxide is dried with steam heat at 100° C. (212° F.). The dry hydroxide contains 50 percent BeO.

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Beryllium Oxide

Beryllium oxide is made from beryllium hydroxide as follows:

(a) One to 2 kg. (about 4 lb.) of beryllium hydroxide is placed in a fire-clay container. Eight of these containers are put on a car, which is moved through a 30-meter- (100-foot-) long gas-fired furnace. The furnace is fired with gas at the entrance end and with gas-air mixture at the middle of the furnace, where the temperature inside the furnace reaches 1,000° C. (1,850° F.). It takes 1 day for a charge to go through the furnace but only 1 hour at 1,000° C. (1,850° F.). The resulting product is beryllium oxide.

Beryllium Chloride

Beryllium chloride is made as follows:

(a) Take 100 kg. (220 lb.) of dry beryllium hydroxide (50 percent BeO) and mix with 60 kg. (120 lb.) of charcoal.

(b) Then take 13 kg. (30 lb.) of this mixture in a kneading machine (Werner-Pfleiderer) with 5 kg. (10 lb.) of tar and 5 liters of water, resulting in a crumbling mixture, which is then

(c) Pressed under 150 atmospheres pressure and extruded in long bars that are broken in briquets (wet).

(d) These briquets are heated for 45 minutes at 1,000° C. (1,850° F.) in the same furnace and same way as described above for beryllium oxide, but when in containers the briquets are covered with charcoal to prevent burning.

(e) Then the briquets are chlorinated in a furnace lined with silica brick, using carbon blocks as electrodes. The capacity of the furnace is 10 to 12 kw., and alternating current is used with 120 or 240 volts at the beginning, which later during the process is changed to more amperes and about 25 volts. The time of the process is 26 to 27 hours. Chlorine gas is then passed through the mixture, and the electric current goes through the beryllium chloride and charcoal mixture only to heat.

(f) The beryllium chloride and chlorine go out into a nickel chamber, where the beryllium chloride condenses and settles at the bottom, while the chlorine gas goes out at the top. From 100-kg. (220-lb.) briquets about 100 kg. (220 lb.) of raw beryllium chloride is obtained.

Purification of Beryllium Chloride

Raw beryllium chloride is heated at 500° C. (950° F.) in a chamber through which hydrogen gas is passed. Nitrogen gas is passed through the chamber before and after the run (hydrogen) to eliminate the entrance of air.

The products are:

Pure beryllium chloride
Beryllium chloride and aluminum chloride
Hydrogen and silicon tetrachloride

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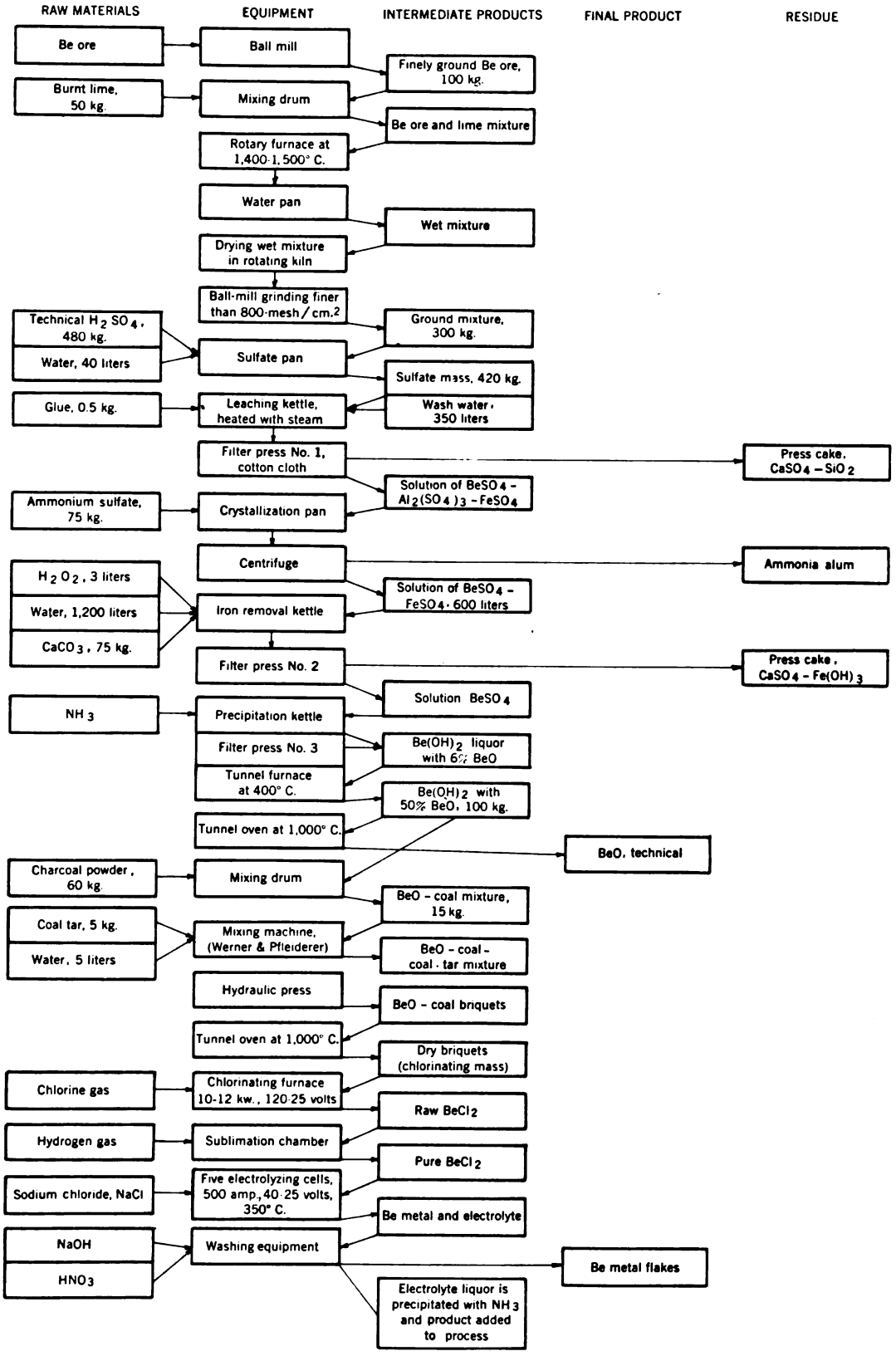
FLOW SHEET FOR BERYLLIUM EXTRACTION



Translated from chart prepared by Degussa (Deutsche Gold und Silver Scheide Anstalt), Frankfurt-am-Main, August 23, 1945.

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In 24 hours 100 kg. (220 lb.) of raw beryllium chloride gives 85 kg. (180 lb.) of pure beryllium chloride.

Beryllium Carbonate (at Rheinfelden)

Wet beryllium hydroxide (6 percent BeO) from the filter press is brought from the Frankfurt plant to the Rheinfelden plant, where 450 kg. (950 lb.) of beryllium hydroxide is mixed with 250 kg. (500 lb.) of ammonium carbonate, 3 liters of ammonium hydroxide, and 500 liters of water and warmed with steam to 50° C. (112° F.) until dissolved; the container is of iron lined with rubber. Then the solution is filtered through the filter press, where aluminum hydroxide is retained.

Sodium sulfide is added to the filtered solution; after standing 24 hours it is filtered to take the iron out as iron sulfide.

The filtered solution is next heated with steam in round enameled pans. The gases NH_3 and CO_2 go out. The remaining beryllium carbonate is filtered in vacuum, beryllium carbonate floating to the top, solution going down.

The beryllium carbonate is washed with water, then dried. Men working with beryllium carbonate have to be replaced every 3 months to avoid development of lung disorders.

Beryllium Nitrate

Beryllium carbonate (50 percent BeO) is dissolved in nitric acid to get beryllium nitrate solution (13 percent $Be(NO_3)_2$), then crystallized cold to crystals of beryllium nitrate.

Beryllium Metal

At Degussa in Frankfurt there are 5 cells (10 containers). One day containers 1, 3, 5, 7, and 9 are operated, and the next day containers 2, 4, 6, 8, and 10.

The fused electrolyte used consists of 50 percent beryllium chloride and 50 percent sodium chloride. Direct current is 500 amperes, with 40 to 50 volts at the start and 25 volts at the end of a 24-hour run; temperature is 350° C. (650° F.). The anode is of graphite and the cathode of nickel.

At the end of a 1-day run the remaining melt in odd-numbered containers is siphoned into the even-numbered containers and vice versa the following day.

Beryllium metal is deposited on nickel in the form of small flakes which grow toward the anode. When the beryllium metal reaches the anode, the chlorine liberated at the anode takes the metal away.

Beryllium metal is removed with a holed spoon. Before the current is turned on to even-numbered containers, more mixture of beryllium chloride and sodium chloride is added. Every 3 to 5 months a new mixture of salts is prepared for electrolysis of beryllium.

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The mixture of beryllium metal and electrolyte is pressed in a round, cylindrical container in ice water. The electrolyte is dissolved out, and the beryllium metal which remains in the container is washed with a large volume of water.

Ammonia is added to the screened beryllium chloride solution to form beryllium hydroxide.

Beryllium metal flakes are washed with sodium hydroxide solution to remove the aluminum and with nitric acid to remove beryllium oxide, then dried in air.

As stated, the new beryllium plant Degussa started to erect at Rheinfelden is only 90 percent completed. There space is provided for only one cell (two containers).

With 5 cells (10 containers) at 215 Guteleutestrasse, Frankfurt, and 1 cell (2 containers) at Rheinfelden, Degussa can produce 210 kg. (410 lb.) of beryllium metal a month.

One ton of beryl yields 20 kg. (44 lb.) of beryllium metal.

Most of the beryllium metal was sold to Heraeus Vacuumschmelze, Hanau, to make alloys containing beryllium.

Production of beryllium metal at the Frankfurt plant of Degussa since 1940 was:

Year	Beryllium metal produced
1940-41	390 kg. (850 lb.)
1942...	610 kg. (1,300 lb.)
1943...	613 kg. (1,305 lb.)
1944...	300 kg. (660 lb.)

USES OF BERYLLIUM

Beryllium metal is used chiefly in metallurgy as a scavenger and as an alloying element or as pure metal for certain parts.

Beryllium oxide is used in ceramics and as a high-temperature refractory. Degussa had developed a sintered beryllium oxide called "Degussit," which is supposed to be gas-tight.

MAKING BERYLLIUM MASTER ALLOYS

Copper-beryllium master alloys were made by Heraeus Vacuumschmelze, Hanau, by reducing beryllium oxide with graphite in the presence of copper powder or copper oxide. The reduction of beryllium oxide is carried out according to the information given by Herbert Gruber, one of the inventors, in his German patents assigned to Heraeus Vacuumschmelze:

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German patent -	Patented -
579,694	1931
588,981	1933
635,701	1935
646,605	1933
663,273	1936
675,050	1933
703,517	
709,740	
722,331	

(a) Copper powder is placed on the bottom of a three-phase Siemens electric-arc furnace. A mixture of beryllium oxide, graphite, and copper oxide is added in small lots. It takes 5 hours for a charge of 150 to 200 kg. (about 400 lb.) of beryllium-copper alloy, the temperature of the furnace being 2,000° C. (3,632° F.).

(b) This makes a copper-beryllium alloy containing 3 to 4 percent Be. There is a loss of 20 percent of the added beryllium.

(c) During cooling, the graphite comes out of the melt. The slag consists of carbon and beryllium carbide. This slag is reused for the next charge.

SPECIAL BERYLLIUM ALLOYS

After many years of research, Heraeus Vacuumschmelze, Hanau, in cooperation with R. Strauman of Waldenburg, Switzerland, has developed beryllium alloys for watch and clock springs and for instruments that surpass in accuracy and performance those made of other alloys. Two main types of alloys have been developed for watch springs: Contracid, analyzing 60 percent Ni, 15 percent Cr, 7 percent Mo, 15 percent Fe, 2 percent Mn, 0.5 percent Si, and 0.5 to 0.75 percent Be, and (2), Nivarox, consisting of four groups of alloys:

	Percent			
Group	CT	M	N	W
Ni....	37.0	31.5	36.0	30.0
Cr....	8.0	-	-	-
Fe....	54.0	60.0	60.0	61.0
Mn....	.85	.75	.80	.70
Be....	.90	.90	.90	.90
Mo....	-	6.50	-	-
Si....	.2	.10	.10	.10
W.....	-	-	-	7.5
Ti....	1.0	-	2.0	-
C.....	Less	than	.10	

The Strauman alloys for watch and clock springs are covered in patents issued to September 11, 1944.

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Nivarox Alloys Patents

Group	M and W	CT	CTC
German.....	578,390	649,811	585,151
Swiss.....	160,798	196,408	166,535 and 227,628
United States	1,974,695	2,072,489	---
British.....	383,174	477,729	---
French.....	735,009	814,143	42,520

BERYLLIUM ALLOY STOCKS

Heraeus Vacuumschmelze at Hanau obtained most of the beryllium metal from Degussa and copper from Metallgesellschaft. As of July 25, 1945, Heraeus Vacuumschmelze had in stock about 18 tons of copper-beryllium alloys, as billets and flats, and about 80 tons of other alloys.

BERYLLIUM LOSSES IN PROCESS

Fron 1939 to 1944 the losses of beryllium at Heraeus Vacuumschmelze were:

	Copper-beryllium master alloys	Beryllium oxide reduction
Remelting scrap	367 kg. (810 lb.)	2,290 kg. (5,040 lb.)
2,910 kg. (6,400 lb.)		
Year	Kg.	Lb.
1939.....	549	1,210
1940.....	771	1,700
1941.....	692	1,522
1942.....	1,185	2,610
1943.....	1,076	2,370
1944.....	1,294	3,950
Total	5,567	13,362

With the increasing demand for production of beryllium alloys by the reduction of beryllium oxide there was an increase in beryllium loss, as may be seen from the following:

Beryllium losses in 1939

	Remelting scrap	Cu-Be master alloys	BeO reduction
Remelting mass.....kg.	93,814	3,646	41,123
Be in charge.....do.	1,993	485	1,195
Be loss.....do.	288	73	188
Be in ingots.....do.	1,705	412	1,007
Be loss from charge..percent	14.5	15.0	15.7
Be loss from ingots.....do.	16.9	17.5	18.6

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Beryllium losses in 1940

	Remelting scrap	Cu-Be master alloys	BeO reduction
Remelting mass.....kg.	114,324	8,476	47,666
Be in charge.....do.	2,511	1,120	1,484
Be in ingots.....do.	2,075	975	1,293
Be loss.....do.	436	145	190
Be loss from charge..percent	17.5	12.9	12.8
Be loss from ingots.....do.	21.7	14.9	14.7

Beryllium losses in 1941

Remelting mass.....kg.	121,104	1,511	50,872
Be in charge.....do.	2,566	192	1,741
Be in ingots.....do.	2,142	171	1,493
Be loss.....do.	424	21	248
Be loss from charge..percent	16.5	10.6	14.2
Be loss from ingots.....do.	19.8	12.0	16.6

Beryllium losses in 1942

Remelting mass.....kg.	161,962	2,538	66,737
Be in charge.....do.	3,482	336	2,251
Be loss.....do.	741	35	409
Be loss from charge..percent	21.3	10.5	18.2
Be loss from ingots.....do.	27.0	11.7	22.2

Beryllium losses in 1943

Remelting mass.....kg.	150,996	6,815	70,101
Be in charge.....do.	3,105	919	2,426
Be in ingots.....do.	2,617	854	1,902
Be loss from charge..percent	15.7	7.0	21.6
Be loss from ingots.....do.	18.7	7.5	27.5

Beryllium losses in 1944

Remelting mass.....kg.	152,814	2,541	83,849
Be in charge.....do.	3,167	374	3,107
Be in ingots.....do.	2,634	346	2,374
Be loss from ingots..percent	20.2	8.2	30.8

BERYLLIUM STEEL

Ruhrstahl A. G., at Witten, has developed (German patent 691,325) a steel containing beryllium; but, as reported by Dr. Völkel of Degussa, this steel was not made in large quantities owing to the shortage of beryllium.

Another German patent (654,829) describes a hard alloy for tools, shells, and bearings and consists of 7 percent Be and 0.50 percent C, with W, Mo, and Cr.

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Stahlwerke Röchling-Buderus A. G. has a German patent (675,871) for sintered metal containing 0.3 to 7.0 percent beryllium.

BERYLLIUM EXTRACTION AND ALLOYS, GERMAN PATENTS

I. G. Farbenindustrie, Frankfurt, has the following German patents:

603,051
705,645
724,119
729,806
741,125

Siemens-Halske, A. G., Berlin, has the following German patents:

603,574
626,402

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